On Demand Analysis: SVOCs by GC/MS
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All samples are extracted by EPA SW-846 Method 3520C followed by analysis using EPA SW-846 Method 8270D.

Analytes
Samples are analyzed for Acenaphthene (CAS # 83-32-9), Acnapthtylene (CAS# 208-96-8), Acetophenone (CAS# 98-52-4), Acetophenone (CAS #98-52-4), Anthracene (CAS #120-12-7), Atrazine (CAS #1912-24-9), Benzaldehyde (CAS #100-52-7), Benzo(a)anthracene (CAS #56-55-3), Benzo(a)pyrene (CAS #50-32-8), Benzo(b)fluoranthene (CAS #205-99-2), Benzo(g,h,i)perylene (CAS #191-24-23), Benzo(k)fluoranthene (CAS #207-08-9), 1,1'-Biphenyl (CAS #92-52-4), bis(2-Chloroethoxy)methane (CAS #111-91-1), bis(2-Chloroethyl)ether (CAS #111-44-4), bis(2-Chloroisopropyl)ether (CAS #108-60-1), bis(2-Ethylhexyl)phthalate (CAS #117-81-7), 4-Bromophenylphenylether (CAS #101-55-3), Butylbenzylphthalate (CAS #85-68-7), Carbazole (CAS #86-74-8), Caprolactam (CAS #105-60-2), 4-Chloroaniline (CAS #106-47-8), 4-Chloro-3-methylphenol (CAS #59-50-7), 2-Chloronaphthalene (CAS #91-58-7), 2-Chlorophenol (CAS #95-57-8), 4-Chlorophenylphenylether (CAS #7005-72-3), Chrysene (CAS #218-01-9), Dibenzo(a,h)anthracene (CAS #53-70-3), Dibenzofuran (CAS #132-64-9), 3,3′-Dichlorobenzidine (CAS #91-94-1), 2,4-Dichlorophenol (CAS #120-83-2), Diethylphthalate (CAS #84-66-2), 2,4-Dimethylphenol (CAS #105-67-9), Dimethylphthalate (CAS #131-11-3), Di-n-Butylphthalate (CAS #87-74-2), 4,6-Dinitro-2-methylphenol (CAS #534-52-1), 2,4,-Dinitrophenol (CAS #51-28-52), 2,4-Dinitrotoluene (CAS #121-14-2), 2,6-Dinitrotoluene (CAS #606-20-2), Di-n-Octylphthalate (CAS #117-84-0), Fluoranthene (CAS #206-44-0), Fluorene (CAS #86-73-7), Hexachlorobenzene (CAS #118-74-1), Hexachlorobutadiene (CAS #87-68-3), Hexachlorocyclopentadiene (CAS #77-47-4), Hexachloroethane -(CAS #67-72-1), Indeno(1,2,3-cd)pyrene (CAS #193-39-5), Isophorone (CAS #78-59-1), 2-Methylnaphthalene (CAS #91-57-6), 2-Methylphenol (CAS #95-48-7), 4-Methylphenol (CAS #106-44-5), Naphthalene (CAS #91-20-3), 2-Nitroaniline (CAS #88-74-4), 3-Nitroaniline (CAS #99-09-2), 4-Nitroaniline (CAS #100-01-6), Nitrobenzene (CAS #98-95-3), 2-Nitrophenol (CAS #88-75-5), 4-Nitrophenol (CAS #100-02-7), N-Nitrosodimethylamine (CAS #62-75-9), N-Nitroso-di-n-propylamine (CAS #921-64-7), N-Nitrosodiphenylamine (CAS #86-30-6), Pentachlorophenol (CAS #87-86-5), Phenanthrene (CAS #85-01-8), Phenol (CAS #108-95-2), Pyrene (CAS #129-00-0), 1,2,4,5-Tetrchlorobenzene (CAS #95-94-3), 2,4,5-Trichlorophenol (CAS #95-95-4), 2,4,6-Trichlorophenol (CAS #88-06-2), 2,3,4,6-Tetrachlorophenol (CAS #58-90-2), 2-Methoxyethanol (CAS #109-86-4), and 1-Methylnapthalene (CAS #90-12-0).

Instrumentation
Agilent 6890/5975 GCMS with a HP-5MS (Agilent Part# 19091S-433) 30 meter by 0.25 mm diameter by 0.25 micron film. The Oven temperature uses an initial temperature of 50 °C and hold 0.5 minutes, ramp at 23 °C/min to 290, and ramp at 15 °C/min to 320 and hold for 4.50 minutes. A constant flow of 1.2 mL/min using pulsed splitless with initial temperature of 250 °C, pressure of 9.78 psi, pulse pressure of 30 psi, pulse time of 0.4 minutes, purge flow of 60 mL/min, purge time of 0.4 minutes, and total flow of 63.8 mL/min. Mass Spectrum is scanned from 35 m/z to 500 m/z with a MS source temperature of 230 °C, and MS Quad of 150 °C.

QC Notes
The following internal standards are used 1,4-dichlorobenzene-d4 (CAS # 3855-82-1), Naphthalene-d8 (CAS # 1146-65-2), Acenaphthene-d10 (CAS# 15067-26-2), Phenanthrene-d10 (CAS# 1517-22-2), Chrysene-d12 (CAS# 719-03-5), and Perylene-d12 (CAS# 1520-96-3). Surrogates used are Phenol-d5 (CAS# 4165-62-2), 2-Fluorophenol (CAS# 367-12-4), 2,4,6-Tribromophenol (CAS# 118-79-6), Nitrobenzene-d5 (CAS# 4165-60-0), 2-Fluorobiphenyl (CAS# 321-60-8), and Terphenyl-d14 (CAS# 1718-51-0).

Acceptable RRFs are 20%.; Other compounds with higher RRFs are Benzaldehyde (40%), bis(2-Chloroisopropyl)ether (35%), Acetophenone (40%), 4-Chloroaniline (40%), Hexachlorobutadiene (35%), Caprolactam (40%), 1,2,4,5-Tetrachlorobenzene (40%), Hexachlorocyclopentadiene (35%), 1,1'-Biphenyl (40%), 2-Nitroaniline (40%), Dimethylphthalate (35%), 3-Nitroaniline (40%), 2,4-Dinitrophenol (35%), 4-Nitrophenol (35%), Diethylphthalate (35%), 4-Nitroaniline (40%), 4,6-Dinitro-2-methylphenol (35%), N-Nitrosodiphenylamine (35%), Atrazine (40%), Carbazole (40%), Di-n-butylphthalate (35%), Butylbenzylphthalate (35%), 3,3'-Dichlorobenzidine (35%), bis(2-Ethylhexyl)phthalate (35%), and Di-n-octylphthalate (35%).

A continuing calibration is run at the midrange of the curve (60 ppb – 40 ppb). Acceptable continuing calibration RPD is +/- 20%. The following compounds are higher than 20%: Benzaldehyde (+/- 40%), Acetophenone (+/- 40%), 4-Methylphenol (+/- 25%), 4-Chloroaniline (+/- 40%), Caprolactam (+/- 40%), 2-Methylnaphthalene (+/- 25%), 1,2,4,5-Tetrachlorobenzene (+/- 40%), 2,4,5-Trichlorophenol (+/- 25%), 1,1-Biphenyl (+/- 40%), 2-Nitroaniline (+/- 40%), 3-Nitroaniline (+/- 40%), 2,3,4,6-Tetrachlorophenol (+/- 25%), Dibenzofuran (+/- 25%), 4-Nitroaniline (+/- 40%), Atrazine (+/- 40%), and Carbazole (+/- 40%).
Limits for surrogate recoveries are Phenol-d5 (10-110%), 2-Fluorophenol (21-110%), 2,4,6-Tribromophenol (10-123%), Nitrobenzene-d5 (35-114%), 2-Fluorobiphenyl (43-116%), and Terphenyl-d14 (33-141%).

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DFTPP acceptance criteria are as follows: Mass 51 30 – 60% of mass 198, Mass 68 Less than 2% of mass 69, Mass 69 present, Mass 70 Less than 2% of mass 69, Mass 127 40 – 60% of mass 198, Mass 197 Less than 1% of mass 198, Mass 198 Base peak, 100% relative abundance, Mass 199 5 – 9% of mass 198, Mass 275 10 – 30% of mass 198, Mass 365 Greater than 1% of mass 198, Mass 441 Present but less than mass 443, Mass 442 Greater than 40% of mass 198, and Mass 443 17 – 23% of mass 442.

Calibration

Initial calibration standard is prepared from Restek Stocks (OLM 01.1 Revised SV Megamix (Cat #31900), Additions Standard (Cat #31902), and N-Nitrosodimethylamine Standard (Cat #31427)) from 5 ppb – 80 ppb (5 ppb, 10 ppb, 20 ppb, 40 ppb, 60 ppb, and 80ppb). Internal standards were added using Restek (Cat# 31206) at 20 ppb. Surrogates were added using Restek B/N Surrogate Mix (Cat# 31062) at 50ppb and Restek Acid Surrogate Mix (Cat# 31063) at 100 pbb.

2-Methoxyethanol (5 ppb – 77 ppb; 5 ppb, 10 ppb, 19 ppb, 39 ppb, 58 ppb, and 77 pbb) and 1-Methylnapthalene (5 ppb – 80 ppb; 5 ppb, 10 ppb, 20 ppb, 40 ppb, 60 ppb, and 80ppb) were prepared in a separate curve using Supleco (Cat# 4-8162) and AccuStandard (Cat# PS-160-01-9766).

A second source was prepared at 60 ppb from Supelco Stocks (TCL Base-Neutrals Mix 1 (Cat #48900-U), TCL Base-Neutrals Mix 2 (Cat# 48120-U), 3,3-Dicholorbenzidine (Cat #48029), TCL Hazardous Substances Mix 1 (Cat# 488907), TCL Hazardous Substances Mix 2 (Cat #48908), TCL Phenols Mix (Cat# 48904), and TCL PAHs (Cat #48904), and EPA CLP SOW OLM04 BNA Mix (Cat #47514-U). No second source was available for 2-Methoxyethanol. 1-Methylnaphthalne second source was from AccuStandard (Cat# H-001S). A second source was not used for 1,2,4,5-Tetrachlorobenzene and 2,3,4,6-Tetrachlorophenol.

Integration Information

Chemstation software adds the letter "m" to indicate a manual integration. The OASQA manual integration checklist is included in the case file.

Extraction

A 1-L aliquot is acidified to a pH of 2.0 and a solution of surrogates (1.0 mL at 100/50 ug/mL) is added before extraction with methylene chloride using continuous liquid-liquid extraction. The extract is concentrated to 1 mL, internal standards are added, and then analyzed by GC/MS.

Matrix Spikes and Blank Spikes

A low spike (5 ppb) and a mid spike (60 ppb) are performed with every set of samples. The Matrix Spike and Matrix Spike Duplicate are spiked at the 60 ppb level. Matrix Spike and Matrix Spike Duplicate requires additional sample volume and is not always provided.

16 compounds plus 2-Methoxyethanol and 1-Methylnaphthalne are reported with the following percent recovery criteria: Phenol 12-110%, 2-Chlorophenol 27-123%, 4-Nitrophenol 10-80%, Pentachlorophenol 9-103%, 4-Chloro-3-methylphenol 23-97%, Benzo(a)pyrene 17-163%, Bis(2-chloroethyl)ether 12-158%, Diethyl phthalate 10-114%, 2,4-Dinitrotoluene 24-96%, Hexachlorobenzene 10-152%, Hexachloroethane 40-113%, Isophorone 21-196%, Naphthalene 21-133%, N-Nitroso-di-n-propylamine 41-116%, N-Nitrosodiphenylamine 30-150%, 4-Chloroaniline 30-150%, 2-Methoxyethanol 30-150%, and 1-Methylnaphthalne 30-150%.

In addition other compounds are examined for acceptable recovery as provided: Acenaphthene 60.1-132.3%, Acenaphthylene 53.5-126.0%, Acetophenone 30-150%, Anthracene 43.4-118.0%, Atrazine 30-150%, Benzaldehyde 30-150%, Benzo[a]anthracene 41.8-133.0%, Benzo[b]fluoranthene 42.0-140.4%, Benzo[k]fluoranthene 25.2-145.7%, Benzo[g,h,i]perylene D-195.0%, 1,1'-Biphenyl 30-150%, Bis(2-chloroethoxy)methane 49.2-164.7%, Bis(2-chloroisopropyl)ether 62.8-138.6%, Bis(2-ethylhexyl)phthalate 28.9-136.8%, 4-Bromophenyl-phenylether 64.9-114.4%, Butylbenzylphthalate D-139.9%, Caprolactam 30-150%, Carbazole 30-150%, 2-Chloronaphthalene 64.5-113.5%, 4-Chlorophenyl-phenylether 38.4-144.7%, Chrysene 44.1-139.9%, Dibenzo[a,h]anthracene D-199.7%, Dibenzofuran 30-150%, 3,3'-Dichlorobenzidine 8.2-212.5%, 2,4-Dichlorophenol 52.5-121.7%, 2,4-Dimethylphenol 41.8-109.0%, Dimethylphthalate D-100.0%, Di-n-butylphthalate 8.4-111.0%, 4,6-Dinitro-2-methylphenol 53.0-100.0%, 2,6-Dinitrotoluene 68.1-136.7%, 2,4-Dinitrophenol D-172.9%, Di-n-octylphthalate 18.6-131.8%, Fluoranthene 42.9-121.3%, Fluorene 71.6-108.4%, Hexachlorobutadiene 37.8-102.2%, Hexachlorocyclopentadiene D-104%, Indeno[1,2,3-cd]pyrene D-150.9%, 2-Methylnaphthalene 30-150%, 2-Methylphenol 30-150%, 4-Methylphenol 30-150%, 2-Nitroaniline 30-150%, 3-Nitroaniline 30-150%, 4-Nitroaniline 30-150%, Nitrobenzene 54.3-157.6%, 2-Nitrophenol 45.0-166.7%, N-Nitrosodimethylamine 30-150%, Phenanthrene 65.2-108.7%, Pyrene 69.6-100.0%, 2,4,5-Trichlorophenol 30-150%, and 2,4,6-Trichlorophenol 52.4-129.2%.